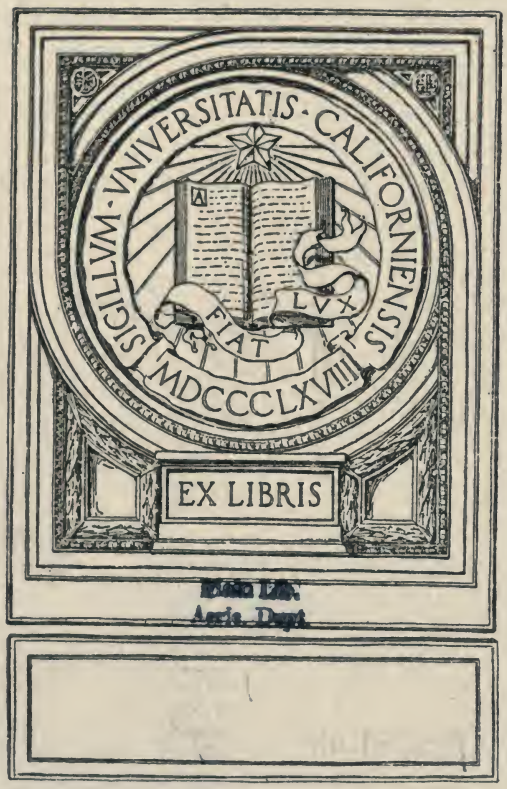

CIRCULAR NO. 8, REVISED.

United States Department of Agriculture,

BUREAU OF CHEMISTRY.

OFFICIAL METHOD FOR ANALYSIS OF TANNING MATERIALS.

[Report of Committee B., A. O. A. C., October, 1902: W. H. Krug, Chairman; C. A. Browne, C. H. Jones, L. S. Munson, H. D. Haskins. Adopted at the Nineteenth Convention of the Association of Official Agricultural Chemists.]

I.—Preparation of Sample.

Barks, woods, leaves, dry extracts, and similar tanning materials should be ground to such a degree of fineness that they can be thoroughly extracted. Fluid extracts must be heated to 50° C., well shaken, and allowed to cool to room temperature.

II.—Quantity of Materials.

In the case of bark and similar material, use such quantity as will give about 0.35–0.45 gram tannins per 100 c. c. of solution, extract in Soxhlet or similar apparatus at steam heat for non-starchy materials. For canaigre and substances containing like amounts of starch use temperature of 50° to 55° C. until near complete extraction, finishing the operation at steam heat. In the case of extract weigh such quantity as will give 0.35–0.45 gram tannins per 100 c. c. of solution, dissolve in 900 c. c. of water at 80°, let stand twelve hours, and make up to 1,000 c. c.

III.—Total Solids.

Shake the solution, and without filtering immediately measure out 100 c. c. with a pipette, evaporate in a weighed dish, and dry to constant weight, at the temperature of boiling water.

IV.—Soluble Solids.

Single pleated filter paper (S. and S., No. 590, 15 cm.) shall be used. To 2 grams kaolin add 75 c. c. of the tanning solution, stir, let stand fifteen minutes, and decant as much as possible (not on the filter), add 75 c. c. of the solution, stir, and pour on the filter. Keep filter full, reject the first 150 c. c. of filtrate, evaporate and dry next 100 c. c. The portion dried for determination shall be perfectly clear and evaporation during filtration must be guarded against.

V.—Non-Tannins.

Prepare 20 grams of hide powder by digesting twenty-four hours with 500 c. c. of water and adding 0.6 gram chrome alum in solution, this solution to be added as follows: One-half at the beginning and the other half at least six hours before the end of the digestion. Wash by squeezing through linen, continue the washing until the wash water does not give a precipitate with barium chlorid. Squeeze thoroughly by hand, and remove as much water as possible by means of a press, weigh the pressed hide powder and take approximately one-fourth of it for moisture determination. Weigh this fourth carefully and dry to constant. Weigh the remaining three-fourths carefully and add them to 200 c. c. of the original solution; shake ten minutes, throw on funnel with cotton plug in the stem, and let drain. Collect this filtrate and filter through folded filter (S. and

S., No. 590, 15 cm.), returning the first 25 c. c. and adding 2 grams of kaolin if necessary. Evaporate 100 c. c. of the clear filtrate. The weight of this residue must be corrected for the dilution caused by the water contained in the pressed hide powder. The shaking must be done in some form of mechanical shaker. The simple machine used by druggists, and known as the milk shake, is recommended.

PROVISIONAL METHOD.

To 14 grams of dry chromed hide powder in a shaker glass add 200 c. c. of the tanning solution, let stand two hours, stirring frequently, shake fifteen minutes, throw on funnel with a cotton plug in the stem, and let drain. Collect the filtrate and filter through a folded filter (S. and S. No. 590, 15 cm.), returning the first 25 c. c. and adding 2 grams of kaolin if necessary. Evaporate 100 c. c. of the clear filtrate.

VI.—Tannins.

The amount of these is shown by the difference between the soluble solids and the corrected non-tannins.

VII.—Testing Non-tannin Filtrate.

Test a small portion of the clear non-tannin filtrate with a few drops of a 1 per cent solution of Nelson's gelatin. A cloudiness indicates the presence of tannin, in which case repeat the process described under V, using 25 instead of 20 grams of hide powder.

VIII.—Provisional Method for the Determination of Total Acidity in Liquors.

Place 100 c. c. of the liquor in a 500 c. c. flask and make up to the mark with water. To 100 c. c. of diluted liquor in a flask with tube condenser add 2 grams of chemically pure animal charcoal. Heat to boiling temperature with frequent shaking, cool, filter, and titrate an aliquot portion with decinormal alkali.

IX.—Method of Drying.

Evaporations shall take place under precisely the same conditions as to contact with steam or with a metallic plate. All dryings shall be made in flat-bottom dishes of at least 6 cm. diameter. All dryings called for after evaporation shall be done by one of the following methods under precisely the same conditions, so that the different residues of each analysis may occupy the same shelves in the drying oven.

1. For eight hours at the temperature of boiling water in steam oven.
2. For six hours at 100° C, in air bath.
3. For five hours at 100° C, in vacuo.

H. W. Wiley,
Chief of Bureau and Secretary A. O. A. C.

Approved:
James Wilson,
Secretary of Agriculture.

Washington, D. C., *December 18, 1902.*

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